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Influences of Yb substitution on the intergrain connections and flux pinning properties of Bi-2212 superconductors



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ABSTRACT

Polycrystalline bulks $Bi_2Sr_{2-x}Yb_xCaCu_{2,0}O_{8+\delta}$ (Bi-2212) with Yb doping content of x = 0, 0.01, 0.02 and 0.05) were fabricated by solid state sintering process. Bi-2212 precursor powders were synthesized by modified co-precipitation process, and Yb₂O₃ powders were added into the precursor powders during the calcination process as dopants. The influences of Yb substitution on the lattice parameter, microstructure and related superconducting properties were systematically investigated. The amorphous components within the grain boundaries were observed with HRTEM, which contributed to the formation of weak links. Therefore, both the decreasing number of porosity and better crystallized grain boundary structure after Yb doping obviously enhanced the intergrain connections. Meanwhile, doping of Yb ions into Bi-2212 matrix also contributed to the enhancement of point pining, thus lead to the improvement of in-field critical current density. Based on the enhancements of both intergrain connections and flux pinning properties, improvement of critical current density was obtained with the optimal doping content of x = 0.02.

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1. Introduction

Since the first discovery of $Bi_2Sr_2CaCu_2O_x$ (Bi-2212), it is considered to be one of the most promising high-temperature superconductors (HTS) due to its excellent properties under lowtemperature, high magnetic field conditions, such as the high irreversibility field beyond 100 T [1] and high critical current density exceeding 266 A mm⁻² up to 45 T [2]. As the only HTS so far, which can be made into round wires with isotropic cross sections, it can greatly simplify the winding process for cables and magnets fabrication. Therefore, it exhibites great potentials for the applications as insert coils in high field magnets and Rutherford cables for accelerators [3–9]. Recently, the maximum total magnetic field of 33.8 T was successfully achieved under the background field of 31.2 T with Bi-2212 insert coils, which proved the effectiveness of Bi-2212 in practical applications.

However, there are two crucial factors which still limit the transport properties of Bi-2212 superconductors. One is the intergrain weak links due to the low extent of texture [10–13], high porosity [14,15] and/or grain boundaries with secondary phases or amorphous layers [16,17]. The other is the weak flux pinning,

which can be attributed to its intrinsic lattice structure [18]. Therefore, optimization processes are required in order to obtain Bi-2212 superconductors with highly orientated microstructure, high density, clean grain boundaries and strong flux pinning properties to enhance the current carrying capacity of Bi-2212 for practical applications. Based on the previous studies, substitution may provide various effects on the optimization of HTS materials by tuning the lattice structure, microstructure, thermal dynamic properties, and electric and/or magnetic properties. Pb substitution at Bi site of Bi-2212 is considered to be the most successful example [19–22]. The introduction of Pb could decrease the lattice parameter c of Bi-2212, which not only reduced the anisotropic behavior of superconducting properties, but also introduced effective flux pinning centers to enhance the in-field current capacity. Therefore, the superconducting critical parameters were effectively optimized. Rare earth (RE) elements doping on the Sr/Ca site of Bi-2212 were also studied [10,23-31]. All the reported RE ions, such as Gd³⁺, Ho³⁺, Yb³⁺ and Eu³⁺ could work as effective pinning centers in the BSCCO system, thus obviously improved the critical current density. Besides, by tuning the thermodynamic properties of Bi-2212, the B₂O₃ doping resulted in the faster growth and better alignment of the Bi-2223 grains, which also improved the critical current density. Therefore, it is interesting to study the doping effects of other elements on Bi-2212 for further improvement of superconducting properties.



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In this study, Yb was chosen as dopant to substitute Sr ions in Bi-2212 matrix. It was found that after proper amount of Yb doping, bulks with better orientation and higher density were prepared, which lead to the formation of well crystallized grain boundaries. The influences of Yb ions on the related superconducting properties including weak link behavior and critical current density were systematically investigated.

2. Experimental details

Bi₂Sr_{2-*x*}CaCu_{2.0}O_{8+δ} (*x* = 0,0.01,0.02,and0.05) precursor powders were prepared by modified co-precipitation process [24] with starting materials of Bi₂O₃, SrCO₃, CaCO₃, and CuO (>99.9%). Yb₂O₃ (>99.99%) powders with the atomic ratio of 0, 0.01, 0.02 and 0.05 corresponding to the *x* value were added before the calcination process. Single phased Yb doped Bi-2212 precursor powders were obtained after a series of calcination processes in air at 800 °C/12 h, 820 °C/20 h, and 850 °C/20 h with intermediate grinding. Silver crucibles were adopted for the calcination in order to avoid the contamination of powders. The precursor powders were then densely packed into a stainless steel die and cold pressed into pellets with the diameter of Ø30 mm, and thickness of 1.5 mm. The bulks were then sintered at 865 °C for 24 h in ambient atmosphere.

The density of bulks was measured using the standard Archimedes method. Polycrystalline X-ray diffraction (XRD) patterns on both precursor powders and bulks were taken on an X-ray diffraction (XRD, Bruker D8 Advance) with Cu-K α radiation ($\lambda = 0.1542$ nm). The texture degrees of (001) peaks were mostly used to assess the quality of textural structures, which were calculated as following,

$$F_{00l} = \frac{\sum I_{00l}}{\sum l} \times 100\% \tag{1}$$

where I_{00l} represents for the intensity of (00l) diffraction peaks of Bi-2212, $\sum I$ is the total diffraction intensity of the pattern. Transmission electron microscopy (TEM) samples were prepared by a grinding dimpling, and ion milling (Gatan PIPS). The high-resolution transmission electron microscopy (HRTEM) was performed on a JEOL JEM-2010 microscope. The scanning electron microscopy (SEM) of fracture surface and backscattering electron (BSE) images were obtained with JEOL JSM-6700F. The compositional analysis was taken by Inca-X-Stream energy-dispersive X-ray spectroscopy (EDX). The AC susceptibility was measured by the Superconducting Quantum Interference Device (SQUID, MPMS-XL-7) with the AC magnetic field of 0.1, 0.5, 1.0 and 1.5 Oe and frequency of 333 Hz. Meanwhile, SQUID was also used to measure the magnetic susceptibility with the background temperature of 4.2 K. The same specimen was used for both the AC susceptibility and magnetization measurements. The specimens were cut from the pellet with the dimension of $\sim 2 \times 2 \times 1.5$ mm, with the 2 \times 2 surface parallel to the pellet surface. During the measurements, specimen was put into a capsule with the 2×2 surface perpendicular to the magnetic field direction and fixed with low-temperature glue to avoid the movements of specimen in magnetic field or during cooling. After that, Bean model [32] was adopted for the calculation of the critical current density (J_c) as shown below,

$$J_{\rm c} = \frac{20\Delta M}{b(1 - b/3a)} \quad (b < a) \tag{2}$$

where *a* and *b* are the length and the width of the specimen respectively, which are both perpendicular to the applied magnetic field, and ΔM is the difference in magnetization between the magnetization value with increasing field M^+ and decreasing field M^- at same magnetic field.

3. Results

The X-ray diffraction patterns of precursor powders after calcination are shown in Fig. 1(a). The major phase can be indexed into orthorhombic Bi-2212 structures with no detectable secondary phase. The X-ray diffraction patterns of the sintered bulks are plotted in Fig. 1(b). After sintering, the major phase is still Bi-2212 in all these bulks. Diffraction peak of the secondary phase is undetectable in the sintered bulks. And the diffraction peak of Yb₂O₃ can be observed only on the x = 0.05 sample. Besides, textural structures can also be noticed with the obvious increasing intensity of (001) peaks, comparing with Fig. 1(a).

As shown in Fig. 2(a) and (b), the SEM images of fractured surface of the x = 0 and 0.02 bulk are obtained. Misoriented grains with small average grain size can be observed on the Yb free bulk. While after Yb doping, the plate-like grains with larger radius and similar thickness are obtained. And the texture with the plate surface perpendicular to the pressing direction is formed.

Generally speaking, there are usually some secondary phases appeared, including Bi-2201 (Bi₂Sr₂CuO_{6+ δ}) and alkali earth cuprates (AEC, (Ca, Sr)_mCu_nO_z) in the sintered Bi-2212 bulks. The BSE images of *x* = 0 and 0.02 bulks are shown in Fig. 2(c) and (d), respectively. By combining the EDX analyses, phase distribution can be distinguished in the backscattering images. The gray background represents the Bi-2212 major phase, white particles are Bi-2201 and black dots are AEC phases and pores. By combining the BSE images and secondary electron images, the AEC phase and pores can further be distinguished and most of the black dots are determined to be pores. The area ratio of Bi-2201 phase in Yb-free and *x* = 0.02 bulks are ~4.8% and ~2.2%, respectively. And



Fig. 1. (a) X ray diffraction patterns of Yb doped Bi-2212 (a) precursor powders and (b) sintered bulks.

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